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3D metaphotonic nanostructures with intrinsic chirality

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Abstract: Chirality is a universal geometry property in both micro- and macro-worlds. Recently, optical chiral effects have drawn increased attention due to their great potential in fundamental studies and practical applications. Significantly, the optical chiral response of artificial structures can be enhanced by orders of magnitude compared to their naturally occurring counterparts. These man-made structures generally exhibit two types of optical chirality: extrinsic chirality and intrinsic chirality. The former relies on the external illumination conditions, while the later arises from the geometric characteristics of three-dimensional objects. In this Review, we mainly focus on the intrinsic chirality of artificial

structures and discuss the existing realizations based on their design principles. In particular, we give an overview of the recent demonstrations of nonlinear optical effects in chiral structures and active chiral structures. At last, we outline some promising prospects for future studies in the field.

1. Introduction

Chirality is a geometrical property of an object, which can be found universally in macro-structures such as human hands and micro-structures such as molecules. A chiral object cannot be superposed onto its mirror image via either translation or rotation operation, or via both. A chiral object and its mirror image are called enantiomorphs or, when referring to molecules, enantiomers, which are often labeled by “left handed” or “right handed”. It has already been noticed that macroscopic chirality ubiquitously exists in various geometric structures, for instance, the rotation of seashells and the growth sequence of plant leaves. Many daily items also utilize and benefit from chirality, including keys, staircases and ropes. In fact, mirror symmetry is much easier to be broken than to be maintained, so it is more common for an object being chiral. Chirality holds important position in fundamental physics because it is induced by symmetry breaking. For example, chirality has become an important property of micro-particles[1] after the proposition of non-conservation of parity.[2] However, chirality was firstly noticed as an essential material property in modern science for the fact that organic molecules with different chirality possess distinct chemical behaviors. As the most essential organic molecules in living organisms, amino acids necessarily have chirality; they are composed of four different chemical groups and a carbon atom attached with each of them, which is also the most explicit definition of chiral molecules. In living organisms, all 21 essential amino acids are L-enantiomers, indicating high selection pressures for chirality resulted from life-maintaining chemical reactions.[3] The geometric structure of a molecule determines its optical chirality. However, it is very challenging to determine the geometric

structure of molecules directly with current technologies. Most molecules have different optical responses, such as refractive index and absorption, when illuminated by circularly polarized light (CPL) with different polarities. Therefore, the optical chiral response of molecules can be a promising indirect approach to distinguish their geometric information.

The outstanding properties of optical chiral materials mainly arise from its different responses to left circularly polarized (LCP) and right circularly polarized (RCP) light. When a linearly polarized light passes through a chiral material, its plane of polarization will rotate by a certain angle due to the difference in refractive index for LCP and RCP light. This effect is so-called optical rotation (OR), or circular birefringence (CB). On the other hand, the difference in absorption of LCP and RCP light is usually called circular dichroism (CD).[4] OR and CD are basic representations of optical chirality. In contrast, linear birefringence (LB) and linear dichroism (LD) are caused by the different refraction indices and absorption coefficients for the two linearly polarized waves with orthogonal directions, respectively, which arise from the anisotropy of structures.[5,6] Both responses to circularly polarized light and linearly polarized light could be complementary techniques to elucidate the structure information of biological or chemical molecules. However, when detecting the structures of molecules and nanostructures, LB and LD cannot be found in solutions where the direction of the molecules or nanostructures are random, while OR and CD have no such requirement.

Recently, OR and CD measurements are widely used to detect the structural, kinetic, and thermodynamic information of molecules, especially to analyze the secondary structure and conformation of macromolecules.[7] However, the optical chiral response of most molecules is hard to be detected, which mainly results from their weak optical response, low concentration and random orientation in solution.

Optical chirality will not only be generated by the quantum responses of molecules, but also be constructed by nanostructures. Furthermore, particular nanostructures could give optical chirality much stronger than molecules. This kind of special nanostructures could be

found in living organism. For example, gyroid nanostructures produce the vivid colors of butterfly wings,[8] and chiral microstructure patterns make jeweled beetles more brilliant under LCP illumination than under RCP illumination.[9]

Recent studies on the optical response of artificial nanostructures, especially plasmonic metallic nanostructures, have promoted breakthroughs in the design of optical chiral materials. In a plasmonic system, the free electrons of metal can collectively oscillate at a dielectric/metal interface. When the charge oscillation is coupled with an incident electromagnetic radiation, the excited surface plasmon polaritons (SPPs) can propagate along the interface. On the other hand, when the charge oscillation occurs in a finite three-dimensional (3D) nanostructure, it generates a standing wave and gives rise to a resonance, called a localized surface plasmon resonance (LSPR). For both types of surface plasmons (SPs), the electromagnetic fields can be tightly confined along the direction normal to the structure surface, and then can generate large near-field enhancement as well as strong far-field optical response. Furthermore, such resonances are mainly determined by the motion patterns of electrons inside. Various optical responses, such as perfect absorption[10,11] and phase modulation,[12] can be achieved with plasmonic nanostructures of different size, shape and geometry. In addition, by virtue of the strong near fields, the plasmon resonances of individual nanoparticles in a cluster can couple with each other and form collective modes based on their spatial arrangement. The plasmon hybridization theory was developed to describe the coupling behaviors between LSPR modes and the generation of new plasmonic modes, by considering the hybridization of individual modes.[13] The past decade has witnessed a tremendous interest in such nanoclusters for the creation of unusual optical properties and extended LSPR modes with new optical properties as compared to isolated nanoparticles.[14]

There are many theories successfully describing the optical chiral response of molecules. The simplest approach is to describe the chiral molecule by a single electron on

helix,[15,16] which can predict the OR and CD response of some special kinds of molecules. For more common cases, we can treat the optical response of one molecule or one part of the molecule as an oscillator, and construct a coupled oscillator model.[17–22] This model could describe the optical chirality of complex molecules. These theories told us that it usually needs strict requirements for generating strong optical chiral signals, even with chiral geometry. In addition, the geometry and molecular states of biochemical molecules are hard to be changed. Fortunately, the development of metamaterials provide a novel platform for designing optical chiral materials with strong and adjustable signals through shaping composited structures and their arrangement. Excitingly, artificial resonant modes such as LSPRs could be used to mimic the quantum oscillators in molecules, and the coupling between LSPRs may generate optical chirality orders of magnitude enhancement in comparison with the counterparts of molecules. Mature fabrication methods such as photolithography and electron-beam lithography (EBL), could easily realize planar structures with the mirror symmetry along the normal direction of the sample surface. Whereas, it is difficult to realize 3D chiral nanostructures without mirror symmetry along any direction. An intuitional way to break this symmetry is to fabricate double-layered nanostructures. In addition, many other special methods have also been developed to fabricate 3D chiral nanostructures. As such, the chirality of molecules can also be enhanced by orders using achiral nanostructures *via* a strong localized near field.[23–29] This effect is very useful in biomolecule sensing.[30,31]

On the other hand, planar structures also could have OR or CD response under an oblique illumination, which is called extrinsic chirality, separated with the intrinsic chirality – the chirality of 3D chiral structures.[32–34] To avoid the influence of extrinsic chirality, nanostructures was always forms four-fold (C4) clusters, in which the extrinsic chirality of each structure would cancel each other, and the chiral signal here would be purely the intrinsic chirality.[35–39] When the nanostructures are in solutions, we also do not need to

consider the extrinsic chirality, for they are randomly distributed and then the overall system must have no anisotropy. In this review, we only focus on the intrinsic chirality generated by the 3D chiral structures, for it is more stable and universal, just like the chirality of molecules.

This review article is organized as follows. We will first discuss the design principle of chiral nanostructures for producing optical chirality. We will then review relevant fabrication methods realizing the chiral nanostructures and discuss the applications of chiral nanostructures, including second-harmonic generation (SHG), enhanced light–matter interactions and active chiral materials. Finally, we will provide a conclusion and a brief perspective on the future development of artificial chiral optical materials.

2. Design principle of optical chiral nanostructures

Geometric chirality and optical chirality are not equivalent to each other. Symmetry analysis can help to recognize the geometric chirality, but it cannot predict the optical chiral response. In fact, chiral structures do not necessarily have optical chiral response, neither OR nor CD, not even to mention an enhanced chiral response. For an optical chiral medium, the constitutive relation can be expressed as[40–42]

$$\vec{D} = \varepsilon_0 \overleftrightarrow{\varepsilon}_r \vec{E} + \frac{i\overleftrightarrow{\chi}}{c_0} \vec{H} \text{ and}$$

$$\vec{B} = -\frac{i\overleftrightarrow{\chi}}{c_0} \vec{E} + \mu_0 \overleftrightarrow{\mu}_r \vec{H}, \quad (1)$$

where ε_0, $\mu_0$and c_0 are the permittivity, permeability and light speed in vacuum, respectively; $\overleftrightarrow{\varepsilon}_r$, $\overleftrightarrow{\mu}_r$ and $\overleftrightarrow{\chi}$ are the relative permittivity, permeability and chirality tensors, respectively. It is obvious that a material with optical chiral response needs electric and magnetic fields coupled to each other. To achieve nanostructures with an intense optical chiral response, we need to carefully investigate the following question from a fundamental principle level, *i.e.*, what are the basic features of structures supporting different responses to their electric and magnetic field under LCP and RCP illuminations. For simplicity, we consider the case that the material

is isotropic, which means the parameters used in **Equation 1** are scalars. Therefore, the refraction index and impedance can be expressed as[40–42]

$$n_{\pm} = \sqrt{\varepsilon_r \mu_r} \pm \chi \text{ and}$$

$$z_{\pm} = z_0 \sqrt{\mu_r / \varepsilon_r}\,, \tag{2}$$

where + is for RCP and – is for LCP; z_0 is the vacuum impedance. If we consider a chiral material with a thickness of d, the phase difference between the LCP and RCP waves is $\mathrm{Re}(n_+ - n_-)dk_0$, where k_0 is the wave vector in vacuum. Thus, the OR can be expressed as $\theta = Re(\chi)dk_0$. Similarly, we can express the transmittance as $T_{\pm} = \exp[-\mathrm{Im}(n_+ - n_-)k_0 d]$. In case of weak absorption, CD can be further simplified as $CD \approx 2k_0 d\mathrm{Im}(\chi)$.

Equation 2 suggests one possible realization of negative refraction. When the chirality of the material is strong enough, *i.e.*, $\mathrm{Re}(|\chi|^2) \gg \mathrm{Re}(\varepsilon_r \mu_r)$, the material will present a negative refractive index for LCP or RCP light, which has been theoretically proposed by Tretyakov *et al*,[43] discussed by Pendry[44] and Monzon, *et al*,[45] and experimentally verified in microwave,[46–48] terahertz (THz)[49] and optical regions.[50,51]

Equations 1 and **2** give us a hint that strong optical chiral response can be generated via a coupling between enhanced electric and magnetic responses [32-35]. Therefore, the question becomes how to generate an electric resonance and a magnetic resonance. That is exactly what artificial nanostructures can provide. By judiciously designing the geometric features of nanostructures, strong electric field and magnetic field can be generated through strong localization of EM field at subwavelength space, which can mimic the oscillations of molecules. Therefore, those structures can be termed as meta-atoms or meta-molecules. By a way of contrast, the resonances of nanostructures can be manipulated artificially. Nevertheless, further demands need to be fulfilled. For example, split ring resonators (SRRs), as the widely used building blocks for metamaterials, support both electric and magnetic resonances. However, no chirality takes place because of orthogonality of two resonances,

which results in no coupling. Therefore, how to make electric and magnetic resonances couple with each other efficiently becomes a crucial requirement to generate optical chiral responses. To introduce a parallel component between two resonances, helix metal structures were proposed by Engheta *et al*.[40] As shown in **Figure 1a**, a helix gold nanostructure with only one round supports two types of resonant modes when illuminated by CPL coming from the direction parallel to or vertical to its rotation axis. When a CPL is incident along the direction vertical to the axis, both electric dipole and magnetic dipole could be excited with well-aligned directions at its fundamental resonance at about 1080 nm, as shown in the right panel of **Figure 1a**. Our simulation results (red line in **Figure 1b)** verify that strong CD response can be observed at the resonances supported by the helix structure under this condition. We can see the lowest three LSPRs at 1080, 750 and 680 nm, and each of them exhibits a CD peak.

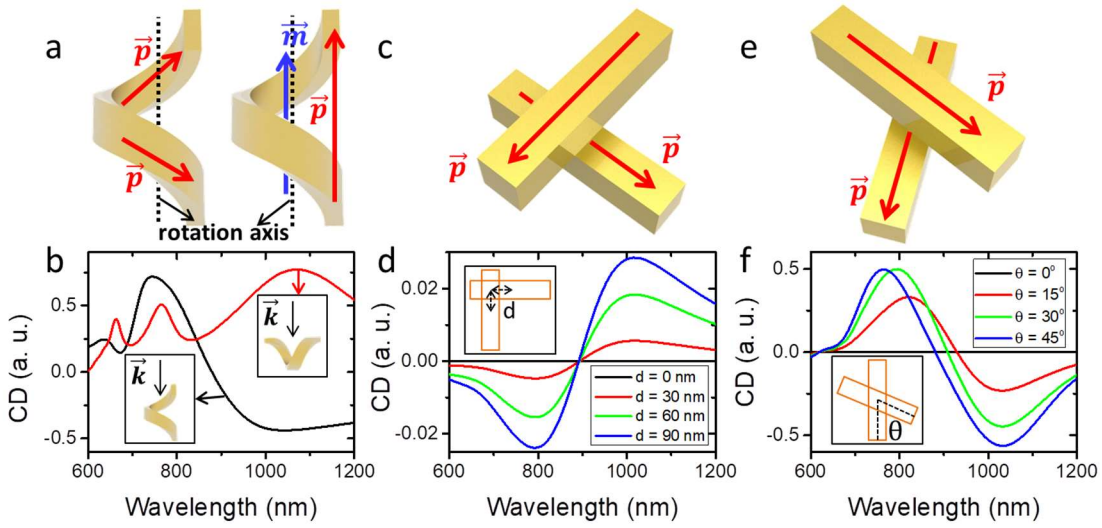

Figure 1. Typical optical chiral nanostructures and their CD spectra. (a) The schematic of the electric and magnetic dipoles in a single helix nanoparticle at its lowest (right) and second lowest (left) resonances, and their corresponding (b) CD spectrum when the incident wave is parallel (black line) and vertical (red line) to the axis of the helix. (c) The schematic of two stacked gold nanobars orthogonal to each other, and its simulated CD spectrum when the centers of the bars shift a distance of d. (e) The schematic of two gold nanobars nonorthogonal to each other, and its (f) simulated CD spectrum when changing the angle between the two nanobars. In FDTD simulations, periodic boundary conditions were used with a lattice constant of 400 nm. The system is illuminated by normal incidence and the

background is vacuum. All the nanostructures in simulations are made of gold from the Palik database. The helix has a 60 × 100 nm^2 cross section and a diameter of 230 nm with a total length of 380 nm. The size of gold nanobars is 60 × 60 × 300 nm^3. The distance between the two nanobars along the direction of light is 200 nm

However, it is still challenging to fabricate helix structures with rotary and out-of-plane features working at visible range. Such a high requirement results from the relative direction between a magnetic dipole and an electric dipole in structures. In general, the directions of two types of dipoles tend to be vertical to each other, which lead to a negligible coupling between two modes. Alternatively, the role of the magnetic dipole can be readily replaced by a specific electric dipole. When a light is incident along the z-axis, a magnetic dipole with a particular magnetic conductivity in x-y plane will have exactly the same response with an electric dipole with the same electric conductivity at a distance of λ/4 along the z-axis. It indicates that an electric resonator placed at one certain plane along the incident direction (called an equiphase plane) is able to mimic a magnetic response at another equiplase plane. For example, as shown in **Figure 1c**, two orthogonal gold nanobars with a small separation along the incident direction can mimic one electric and one magnetic dipole in the same direction. In order to obtain a strong optical chiral response, an efficient coupling between two dipoles can be introduced through either a physical connection or a near-field interaction. Here, taking into consideration of the electric field distribution of the dipole mode on the bars, we can control the coupling strength by varying their center-to-center separation, as shown in the inset of **Figure 1d**. Null CD is observed when two centers overlaps, *i.e.*, d = 0, which results from zero-field at the center of the rod. In contrast, when the ends of two bars gets closer to each other, a strong coupling occurs and the optical chiral response becomes enhanced. The underlying mechanism has be theoretically analyzed.[52,53]

Besides shifting the relative position of the bars, we can also control the coupling strength of two bars by varying their orientations, as shown in **Figure 1e**. Negligible CD is

observed when two bars are orthogonal to each other, *i.e.*, θ = 90°, which is the same as the case of d = 0 in **Figure 1d**. When the angle between two bars decreases, gradually increasing overlap of two near fields will launch the coupling process and then result in an increase in CD signals, as shown in **Figure 1f**. The CD signal approaches the maximum at θ = 45°. Further decrease in the relative angle will make two bars more like a mirror symmetric structure. Then the CD decreases and vanishes when the two bars get parallel.

Especially, when the incident light propagates along the rotation axis, the CD response at the second lowest resonance at ~750 nm in the left panel of **Figure 1a** is stronger than the counterpart supported by the lowest resonance (~1080 nm) or the third lowest resonance (~680 nm), as the black line shown in **Figure 1b**. Interestingly, their signs are opposite. Here, the magnetic dipole generated by the upper and lower parts of the helix would cancel each other mostly due to their opposite current directions. By analyzing this mode, two coupled electric dipole resonators with different directions and in different equiphase planes were observed, similar to the case shown by **Figure 1c** and **1e**. The similar explanation was also be proposed by Gansel *et al*.[54] By comparing the two cases in **Figure 1b**, we can further understand that the optical chirality of one structure is not equivalent to the geometric chirality, since it is also dependent on the excitation conditions.

In brief, previous realizations of strong optical chiral response are designed mainly based on two principles as discussed above, *i.e.*, construction of either nonorthogonal oriented electric and magnetic resonances or two nonparallel electric resonances at different equiphase planes of the incident light. Two types of resonant modes are then coupled with each other through a physical connection or near-field. Interestingly, the helix structure could match both design principles determined by the illumination conditions. Most optical chiral nanostructures can be categorized into these two cases, and we will show it in the next part of this review. Various nanofabrication technologies have been developed to construct these complex structures, including multi-step lithography, direct laser writing (DLW), glancing

angle deposition (GLAD), self-assemble and so on. However, it is not easy to realize 3D nanostructures matching the above requirements. These methods will also be introduced in the next part of this review.

3. Realization of optical chiral nanostructures

3.1. Chiral nanostructures realized by electric-magnetic coupling

Following **Equation 1**, an optical chiral nanostructure can be realized when it supports both electric and magnetic dipoles, which are nonorthogonal to each other and can interact with the incident light efficiently. The original ideal was proposed by Engheta *et al* in 1988,[40] and then realized at microwave range benefiting from the high-speed developing metamaterials in the past ten years.[55–59] These designs are modeled based on a twisted Ω-shaped particle, as shown in **Figures 2a-2b**.

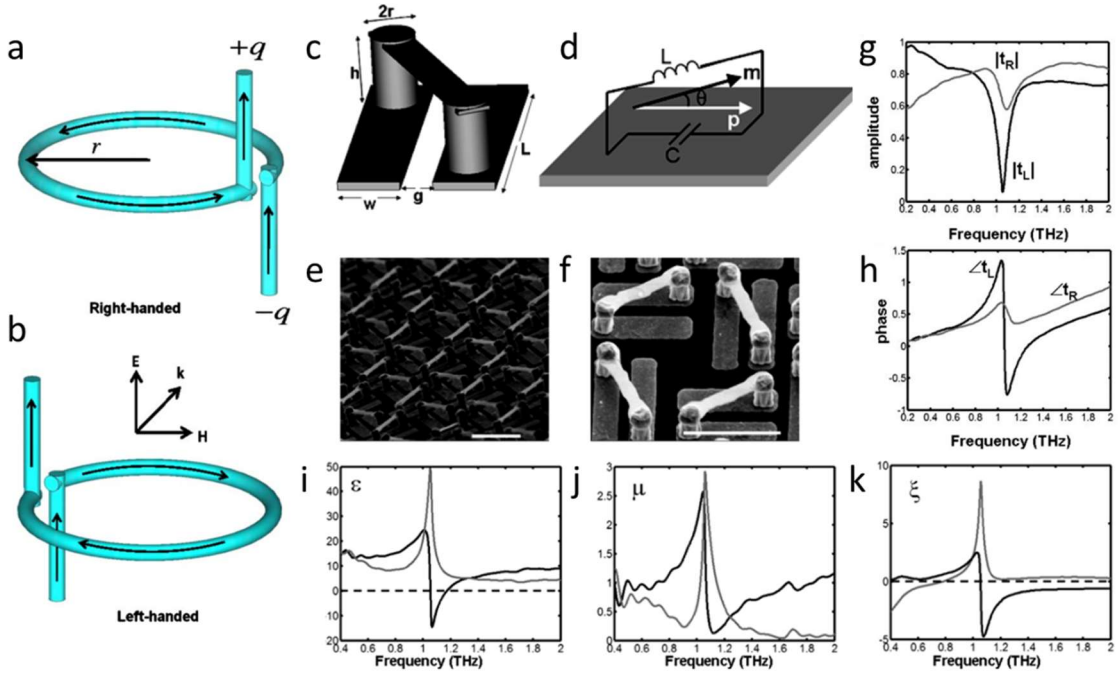

Figure 2. Optical chiral nanostructures realized by coupling of electric-magnetic resonances. (a-b) The schematic of Ω-shaped particles. (c-d) The schematic of the vertical metallic chiral resonator and the direction of its electric and magnetic dipoles. (e-f) SEM images of the experimental sample. (g-h) The measured transmittance and transmission phase of the sample. (i-k) The retrieved effective permittivity, permeability and ξ.
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Subsequently, various multi-layered metallic structures having both electric and magnetic dipoles along the same direction were designed.[55–60] However, most of them are too complicated to be fabricated in micro- or nano-scale. In contrast, it is easier to be realized for THz wave due to the larger structure size.[49] As shown in **Figures 2c-2k**, a rotated split ring was fabricated in a two-layered system. In the lower layer, a capacitor is formed by two rectangular patterns, which act as two electrodes and support two electric dipoles vertical to the capacitor gap. A bridge, nearly parallel to the capacity gap, connect two electrodes and support a magnetic dipole moment along the bridge direction, as schematically shown in Figure 1d. Due to the small angle between the electric dipole and magnetic dipole, a strong coupling could be anticipated. By following the standard effective media,[61,62] the retrieved effective parameters can well express the mechanism of the optical chiral response. Zhao *et al* have used this model to demonstrate the effect media of multiple kinds of chiral structure.[63]

3.2. Chiral nanostructures realized by direct connection of electric dipoles

As the 'typical' chiral structure, helix structure intuitively matches our understanding of chirality. Plasmonic nano-helix structures under normal illumination have been fully studied by Gansel *et al*.[54] By using DLW, chiral holes were first generated on a type of positive-tone photoresist on an indium tin oxide (ITO) glass substrate, and then filled with gold *via* electrochemical deposition. After removing the resist, an elegant freestanding helix array was fabricated. The helix structure is able to generate a very broadband optical chiral response due to the excitation of multiple resonant modes, as indicated by the simulated current distributions in **Figure 3a**. However, the role of each mode in generating optical chiral response differs. The lowest two modes contribute to the big transmission difference under

illumination with different handedness. With such a broad transmission stop band, this helix structure can be potentially applied as a circular polarizer. Later, to further improve the extinction ratio and bandwidth, tapered helices were developed, as shown in **Figure 3b**.[64] Furthermore, to eliminate the anisotropy, twisted nano-helices were proposed.[65–68] As shown in **Figure 3c**, a triple-helix structure was fabricated to hold the rotational symmetry.[68] As given by **Equation 2**, isotropic chiral system can realize an impedance match with the background media. As a result, all the optical chiral response should be ascribed to the absorption. More importantly, these results further verified the physical understanding of the chirality of nano-helix under normal incidence. In twisted helices, the magnetic field does not contribute to the optical chiral response in the absence of the transverse component. Then the chirality must arise from coupling of misaligned electric dipoles at different equiphase planes of the incident light, which can effectively mimic out a magnetic-like response. Another special helical structure was developed that two helices with opposite chirality were connected together.[69] When the upper helices is illuminated by one CPL, the current will flow to the lower helix. Interestingly, the chirality of radiation will be determined by the chirality of the lower helix, *i.e.*, the opposite CPL. Therefore, this structure can work as a circular polarization converter. Radke *et al* developed another technique to fabricate 3D bichiral structures by combining DLW and electroless silver plating, as shown in **Figure 3d**.[70] They firstly use two-photon femtosecond DLW in a negative-tone photoresist to fabricate a 3D bichiral crystal. Then the dielectric template is fully coated with a conformal silver film *via* electroless plating, due to no external current source being required. This method could be readily extended to fabricate extreme complex structures.

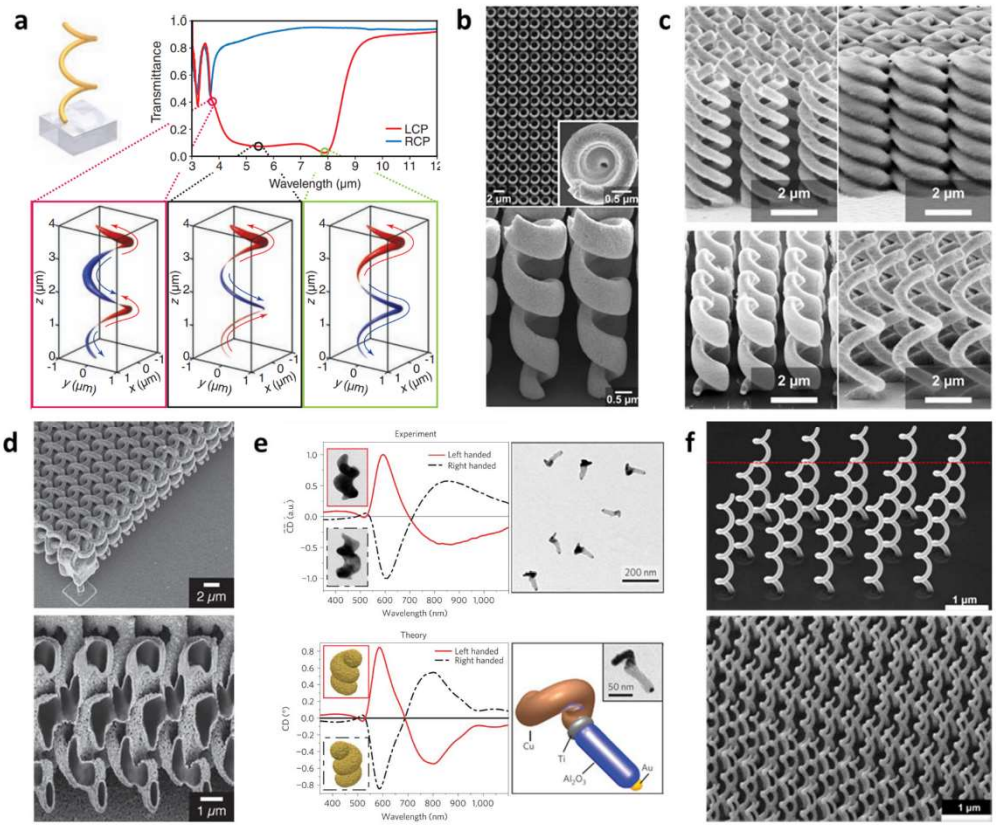

Figure 3. Nano-helices and their optical chiral responses. (a) The simulated transmittance and current distributions of gold nano-helices under illumination of circularly polarized wave. (b) SEM images of the tapered nano-helices. (c) SEM images of nano-helices with different geometric sizes. (d) SEM images of bichiral nanostructures. (e) Measured and simulated CD of nano-spirals, with their SEM images and 3D models. (f) Freestanding metallic spirals fabricated by electron beam-induced deposition.
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Other fabrication methods have also been used to create helices. GLAD was used to fabricate high-quality nano-spirals.[73] Mark *et al* have achieved two-pitched gold nano-helices with a strong optical chiral response around 700 nm.[71] The strong optical chiral response was ensured by the good quality of their structures, indicated by the perfect

matching between the simulation and experimental results shown in **Figure 3e**. Bai *et al* followed this method and fabricated silver nano-helices with optical chiral responses at a shorter wavelength.[74] Electron beam–induced deposition has been used to produce nano-spirals by Höflich *et al*[75], but they cannot well control the quality of their fabrications. Recently, Esposito *et al*[72,76,77] can fabricate nano-spirals with remarkable qualities, which ensured strong optical chiral response in the visible regime by multiple LSPRs, as shown in **Figure 3f**

Although helix structures have been demonstrated as an effective medium to generated optical chiral response, high fabrication cost and limited total fabrication areas still make them ineligible for practical applications. However, in principle, high geometric chirality is not an indispensable component to achieve optical chiral response. In the following sections, we will introduce other works that have reached strong optical chiral responses without perfect helix structures. Especially, the released sample fabrication challenge will greatly enlarge the applications of artificial chiral structures.

One of the major challenge is to realize freestanding helix structures, since it is almost inevitable to include a small connection between structures and the substrate for the consideration of mechanical stability and processing difficulty. If the rotation turn is less than one, the whole circular structure can maintain attached with substrate, which was achieved by Frank *et al* using colloidal nanohole lithography, as shown in **Figure 4a**.[78] Intense optical chiral response was observed based on the hybridization of a series of LSPRs. By improving the fabrication technique for planar nanostructure, many efforts have been developed for 3D structure fabrication with chiral response. It is intuitive to construct a non-planar substrate, preventing the attached structures from intrinsic mirror symmetry along the direction perpendicular to the plane. As shown in **Figure 4b**, for the first time, Dietrich *et al* demonstrated a 3D L-shaped metallic strip with strong chirality *via* excitation of complex LSPRs.[79] By applying similar designs, Dietrich *et al* further fabricated star-shaped structures

on a cone-shaped substrate, and also observed high CD response.[80] Subsequently, many researchers have obtained structures with intense chirality through metallic strip on substrate with particular structures.[81–84] In these studies, both or one of substrate and metal structure were fabricated using lithography. In particular, relatively high precision demand calls for EBL for nanostructure fabrication. However, one major drawback of such a method is the limited fabrication area, usually up to microscale. To solve the problem, Hou *et al* applied self-assembled polystyrene nanospheres as substrate, and multi-step GLAD to construct metal up-layer, to achieve structures with both strong optical chiral response and large area, as shown in **Figure 4c**.[85,86]

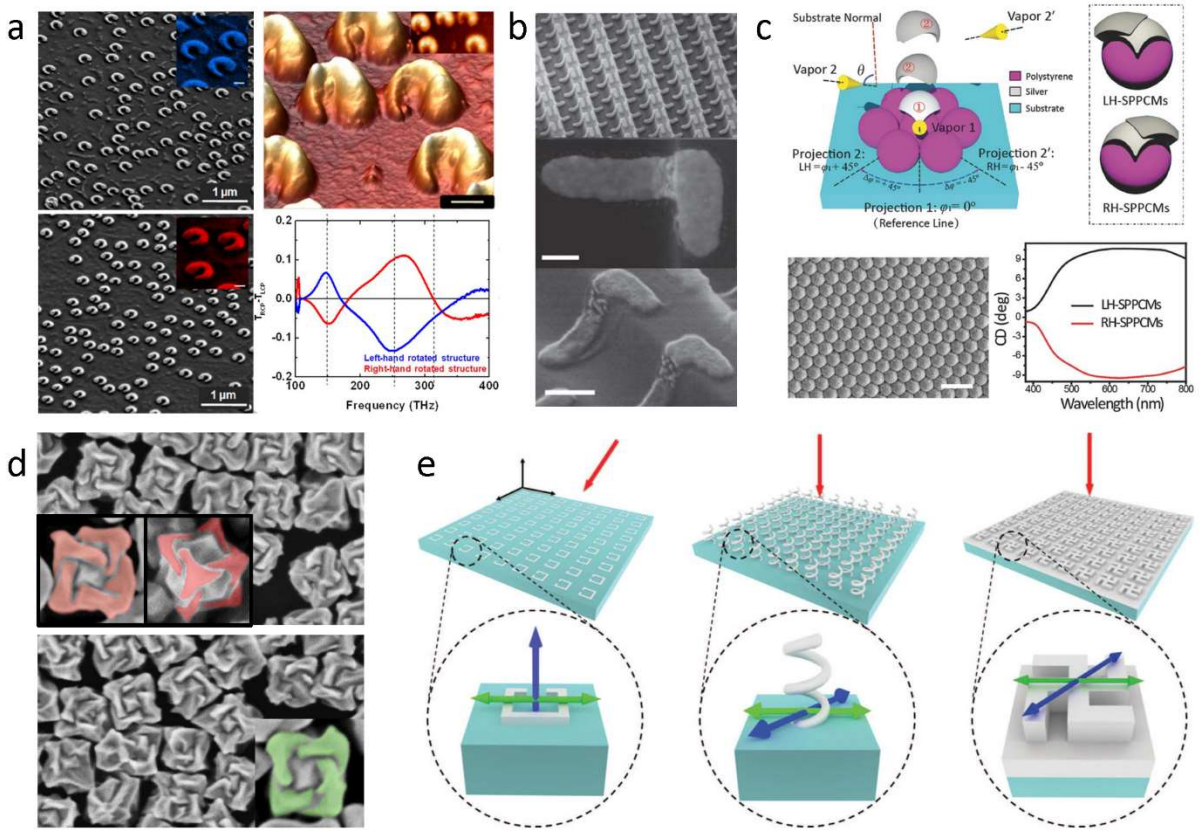

Figure 4. Chiral nanostructures constructed by single particle. (a) SEM images of plasmonic spirals fabricated via colloidal nanohole lithography and their polarization-dependent transmission spectrum. (b) SEM image of metallic L-shaped structures by lithography on a non-planar substrate. (c) Large optical chiral nanostructure via multi-step glancing angle deposition on self-assembled PS nanospheres. (d) SEM images of chiral nanoparticles growing controlled by cysteine chirality transfer. (e) Schematics of 2D extrinsic chiral structure, 3D helix structure and 3D mirror chiral structure. The last two structures are all have intrinsic optical chirality.

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Some other studies have also successfully realized optical chirality within a single nanoparticle by implementing more complicated methods. For example, Helgert *et al* have applied multi-step EBL to produce metal chiral nanostructures composed of two differently oriented L-shapes placed on different planes.[89] Cathcart *et al* have also obtained chiral nanostructures by a self-assembly approach.[90] Lee *et al* grew gold nanoparticles in the environment of chiral molecules, and the chirality of the molecules transfer to the nanoparticles.[87] Then they get two kinds of nanoparticles using molecules with opposite chirality, as shown in **Figure 4d**.

The aforementioned chiral effects are mainly achieved with single particles. Their optical chirality arises from the coupling electric diploes disposed at different equiphase planes along the incident direction *via* direct connections. Besides, there are also other attempts on single-particle-based chirality. For example, starting with a two-dimensional (2D) structure without planar symmetry, the mirror symmetry perpendicular to the plate can be destroyed using a substrate, such as metal. Following such an idea, Karimullah *et al* have accomplished structures with strong optical chirality.[91] Zhu *et al* have done theoretical and experimental analysis of a 2D spiral structure on a metal film, and compared it with 3D helix and 2D extrinsic chiral structure, as shown in **Figure 4e**.[88]

3.3. Chiral nanostructures realized by near-field coupling of electric dipoles

As mentioned in the first section of this review, other than direct connection, near-field coupling is also an important method to make two electric dipoles along different directions at

different equiphase planes of incident light coupling with each other. In this part, we will discuss chiral structures formed by two or more coupled nanoparticles. Although fabrication methods and different product forms are achieved, the basic mechanism of generating chirality is identical to those of **Figures 1c-1f**.

Apart from lithography on non-planar substrate introduced previously, multi-layered planar structures are also an important way to achieve 3D chiral structures. Generally speaking, chiral nanostructures fabricated by such methods are distinguished from two coupled layers of planar structures with different shapes. Due to the high feasibility, chirality on two-layered metal structures was firstly verified in microwave regime.[46–48,92,93] By slightly breaking the symmetry with different sizes at two layers, Decker *et al* observed a modest optical chiral response.[51] Afterwards, Plum *et al* produced relatively stronger optical chirality through introducing a slightly rotating misalignment. A weak optical chirality was also observed on monolayered structure, where the mirror symmetry along planar surface was broken.[50] In addition, it was found that divergence between two layers also help to avoid symmetry perpendicular to the surface. Further analyses revealed that planar symmetry damage in monolayered structure is not necessary. For example, Decker *et al* improved their samples optical chirality without breaking symmetry.[94] The rotated two-layered cross structures is shown in **Figure 5**a. Liu *et al* achieved an adaptable coupling between two-layered U-shaped nanoparticles by arranging their relative orientation, as shown in **Figure 5b**.[95] Chirality of similar structures was then measured and deeply analyzed by Decker *et al*.[96] In this nanostructure, the resonant mode has both electric dipole and magnetic dipole. The electric dipoles in two layers are not parallel, and their coupling are mainly provided by the magnetic field, for the magnetic dipoles of them are in the same direction. In this work, the structures were rearranged according to C4 symmetry, which showed similar effect to twisted helices, *i.e.*, annihilating anisotropy. According to **Equation 2**, CD response under such condition solely results from different absorptions, but not reflections. Here, CD is

merely the result of structure chirality, which excludes impacts of anisotropy. The major advantages of such structure are that only transmission measurement is necessary when performing quantification and that anisotropy is neglected in theoretical analysis. Besides, chirality can be achieved not only by misalignment of same structures, but also by coupling of two layers of different structures. Menzel *et al* have successfully implemented coupling between bar and L-shaped nanostructures to obtain considerable optical chirality, as shown in **Figure 5d**.[97] Following these studies, many research groups have devoted to accomplish various two-layered optical chiral nanostructures *via* their coupling.[98–100] Other than multi-layer lithography, the similar structure can also be realized by other fabrication methods. For example, a nanostructure constructed by two gold dimers was realized in solution based on self-assemble with DNA molecules by Sun *et al*, which is shown in **Figure 5e**.[101]

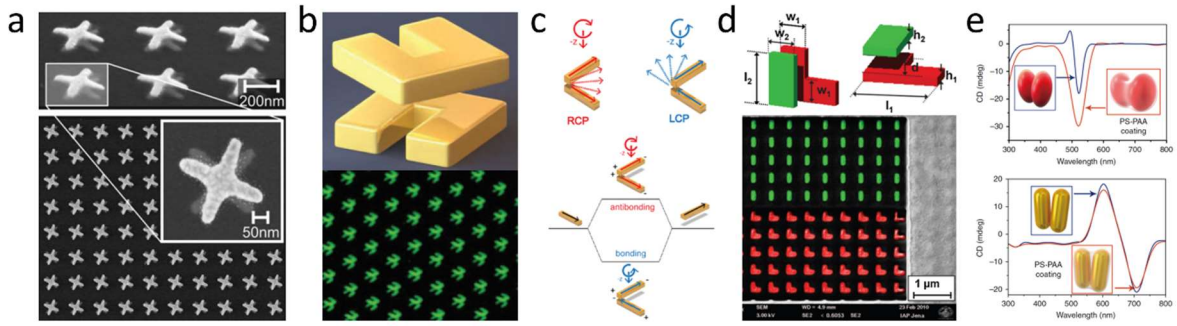

Figure 5. Chiral nanostructures constructed by two coupled particles. (a) SEM images of double layered rotated cross nanostructures.(b) Two U-shaped metal nanoparticles coupled together by the magnetic field vertical to the substrate. (c) Schematics of Born-Kuhn model describing the coupling between two dipoles. (d) Schematics and SEM images of double layered chiral nanostructures by coupling between bar and L-shaped particles. (e) Schematics and CD spectrum of dual-dimmer nanostructure realized by self-assemble in solution.
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As described in the first section, optical chirality of these introduced chiral nanostructures can all be explained by coupling of differently oriented electric dipoles in

different equiphase planes. The difference of coupling behavior here and the structures in last section mainly reside in near-field coupling instead of direct connection. Yin *et al* have used a plasmonic Born−Kuhn model to describe this coupling, schematics of which are shown in **Figure 5c**.[39] Due to the strong coupling between two electric dipoles, two new states, *i.e.*, bounding and anti-bounding states, are generated with different resonance properties, including frequency and amplitude. For most structures, two new states have opposite chiralities, as shown in **Figures 1d** and **1f**. Similar structures can not only be accomplished by lithography, but also by self-assembly process.[102]

Previous works mainly focus on two-layered structures. However, when more layers are involved, the chiral response can be dramatically enhanced due to longer interaction length between structures and the incident light. The corresponding working mechanism can be regarded as an extension of the counterpart of two-layered rotated nanostructures. As shown in **Figure 6a**, Zhao *et al* fabricated a multilayer structure using multi-step lithography and observed a broadband and strong optical chiral response as the number of layers increased.[103] In fact, chirality generating mechanism of this structure was quite similar to that of helix structures. The main difference lay in that near-field coupling dominates here, while direct connection was responsible in nano-helices.

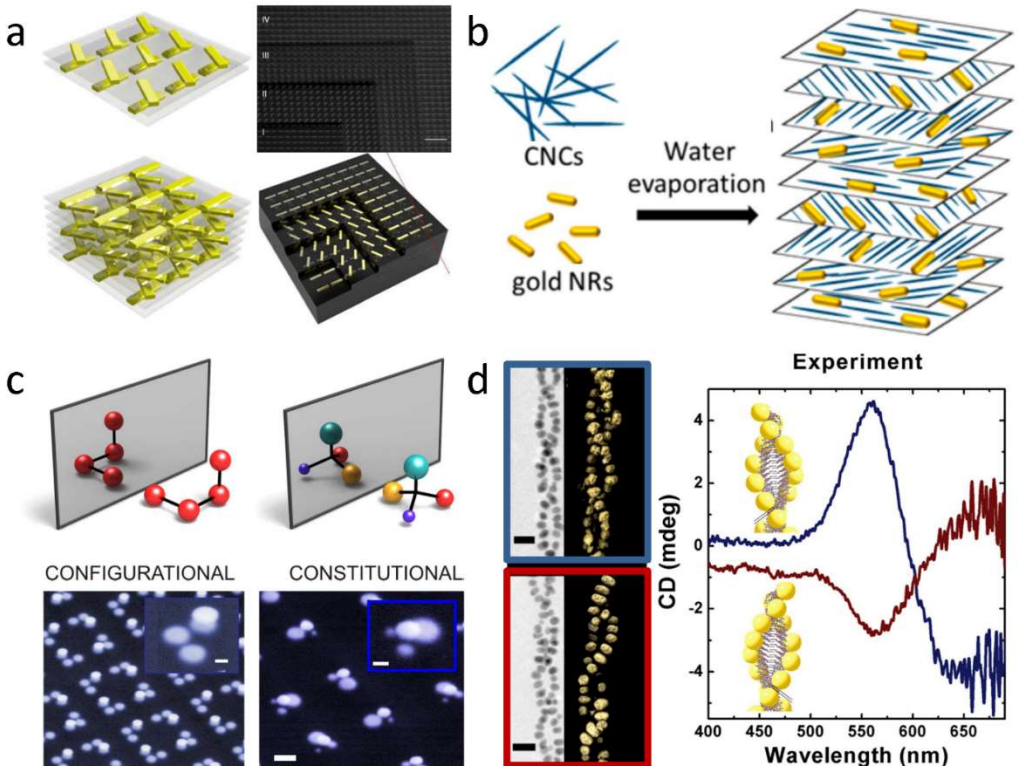

Figure 6. Chiral nanostructures constructed by multiple coupled particles. (a) Schematics and TEM images of multi-layered chiral nanostructure by lithography. (b) Multi-layered chiral nanostructures using self-assembled cellulose nanocrystals with gold nanorods. (c) Four nanoparticles formed chiral nanostructures, based on double-layered lithography. (d) Double helices formed by gold nanospheres. The arrangement of spheres follow the chirality of DNA molecules.
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Reproduced with permission, Querejeta-Fernández et al.[104] (b) Copyright 2014 American Chemical Society
Reproduced with permission, Hentschel et al.[37] (c) Copyright 2012 American Chemical Society
Reproduced with permission, Song et al.[105] (d) Copyright 2013 American Chemical Society

From the perspective of chiral structure fabrication, there are two types of schemes, a direction way, as mentioned in the aforementioned part, and an indirect way, such as self-assemble methods, which generates chiral response by inheriting the chirality from the precursory structures with weak chiral response and enhancing it with strong optical resonances. Querejeta-Fernández *et al* found out that cellulose nanocrystal (CNC) films obtained by water evaporation rotate anticlockwise, forming a left-handed helix. Optical chirality could be obtained when loading with metal nanoparticles.[104,106] Particularly, when the particles are nanorods, their direction will be parallel to the CNCs due to the Van der

Waals force between them, which construct a typical multi-layered chiral structure with electric dipoles rotating along the direction vertical to them, as shown in **Figure 6b**. Similar to cellulose nanocrystals, Lan *et al* applied layered DNA molecules to guide metal nanorods to form into chiral structures,[107] and a similar structure was also achieved by Ma *et al*.[108] However, due to randomness of functionalized structures liquid solution, the final obtained CD signal was not strong enough.

The chiral response in previous works is mainly dependent on the orientation of single particles with directional response. However, for isotropic particles, such as sphere or circular disk, spatial arrangement can alternatively facilitate to generate chiral responses. An innovative theoretical model on 3D chirality structure formed by coupling of multiple spheres was proposed by Fan *et al*.[109] In this structure, chirality is unrelated with single particle shapes, but totally determined by relative position of particles in the entire cluster. Hentschel *et al* firstly verified this idea in a double-layered system, where nano-disks were configured spatially, as shown in **Figure 6c**.[37] However, optical chiral response observed in this study was not significant because that although the structure has satisfied geometric chirality, it is still not qualified for material response requirements. Based on similar ideas, Ogier *et al* obtained a modest chirality using different nano-cylinders in single layer structure.[110]

Self-assembly or other chemical methods were used to obtain similar structures.[111] Kotov *et al* worked out chiral nanostructures with various DNA molecules connected to differently sized nanospheres.[112,113] Shen *et al* obtained similar structures[114] *via* manipulating DNA molecule layer to capture nanospheres. Related theoretical analysis was performed by Ferry *et al*.[115] In this work, DNA molecules was only used as a length tunable connector between nanoparticles. On the other hand, various chemical methods have also been well developed to manipulate complex structures using the intrinsic chirality of DNA molecules. For example, plasmonic nanoparticle self-assembly induced by DNA molecules has become one of the most efficient ways to construct chiral clusters. By utilizing double

helical DNA molecules, Song *et al* obtained nanoclusters with well-defined chiral arrangement, which was verified by the transmission electron microscopy (TEM) image and CD spectrum in **Figure 6d**.[105] The similar techniques were also implemented to realize structures with strong optical chirality by other groups.[116–118] Interestingly, Shen *et al* proposed a rolling-up method by dressing DNA origami with gold nanoparticles to construct 3D plasmonic chiral nanostructures. A planar DNA layer, assembled with well-positioned gold nanospheres, was rationally rolled to form a helix geometry.[119]

4. Applications of optical chiral nanostructures

One of the most important applications of optical chiral nanostructures is to construct optical chiral devices, for instance, the previously mentioned circular polarizer or complex optical field modulator. As introduced in the first section, according to **Equation 2**, negative index can also be obtained by chiral media. Chiral nanostructures actually have much more prospective functions, thus show great potential for practical applications.

4.1. Second harmonic generation in chiral nanostructures

The intrinsic property of chiral nanostructures implies the total absence of mirror symmetry along any directions. In comparison, SHG is only present when the structure has no inversion symmetry, which is a general principle for both molecules and artificial subwavelength structures. Rationally, chiral structures should possess strong SHG property. The relative measurements suggested that optical chiral effect SHG are typically orders of magnitude larger than their linear counterparts.[120–123] The strong SHG signal of chiral structures was firstly verified in planar designs. When an inclined incidence breaks the symmetry of the planar system, optical chiral response, similar to that of 3D chiral structures, would then be generated.[33,124–126] Many groups have conducted SHG studies using extrinsic chirality of

planar structures. Significant SHG emission was observed to confirm that SHG in chiral structures should share the same effect with linear CD, which could be called SHG-CD.[127–130] Nevertheless, there is a difference between extrinsic CD and extrinsic SHG-CD. When the sample is flipped around, the sign of its extrinsic CD would remain, while that of extrinsic SHG-CD would change.[127]

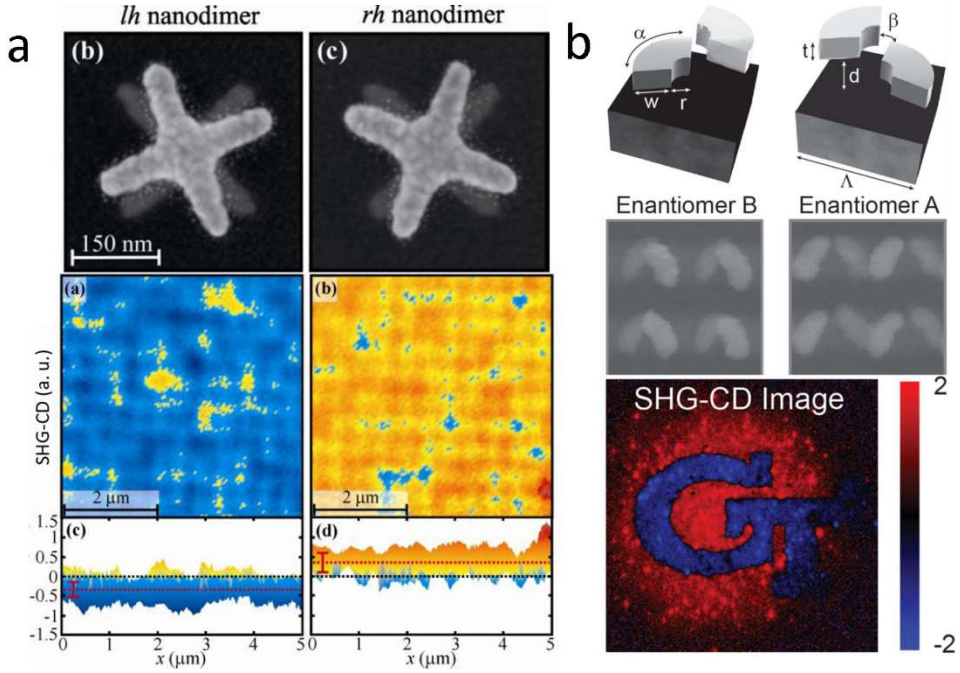

Figure 7. Second harmonic generation CD in 3D chiral structures. (a) TEM images of double-layered chiral nanostructures with C4 symmetry and its SHG-CD image. (b) Schematic and TEM images of double-layered chiral nanostructures without C4 symmetry and its SHG-CD image.
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Huttunen *et al* innovatively observed significant SHG signals of two-layered twisted-cross chiral nanostructures, as shown in **Figure 7a**.[131] Because of C4 symmetry, there is no anisotropy involved. More importantly, SHG signals also have chirality. Inspired by definition of linear CD, the CD of SHG signal can be defined as $\mathrm{SHG\_CD} = 2(P_{LCP}^{2\omega} - P_{RCP}^{2\omega})/(P_{LCP}^{2\omega} + P_{RCP}^{2\omega})$. Structures with opposite chirality will also possess opposite SHG-CD,

indicating that SHG signal generation is closely related to structure chirality. As shown in **Figure 7b**, Sean *et al* replicated the experiment using two-layered chiral structure without C4 symmetry, and revealed that the main mechanism of SHG-CD is chirality rather than anisotropy.[132] SHG signal, in turn, can also be applied to investigate the symmetry information of unknown structures, such as chirality and anisotropy.[41]

4.2. Active optical chiral nanostructures

In principle, chirality of molecule is a fixed property, thus CD spectrum is an important method for material identification and measurement. For specific materials, chirality change always indicates changes in atom arrangements, *i.e.*, chemical reactions. As to artificial materials, its chirality is not determined by atom arrangement inside molecules, but by structural geometry. In consequence, structural chirality can be modulated with more freedom. However, it should be noted that chirality of nanostructures is usually fixed once the fabrication completes. This drawback has motivated further studies on structures with adaptable chirality, because optical chirality is an important and easily measurable property. Thus its active modulation, *i.e.*, changing chirality of existing structures, is of great significance.

There are mainly two routes to regulate optical property of existing structures, *i.e.*, varying the properties and the geometry of involved materials. The former relies on the adaptable material properties, such as dielectric constant, which can be modulated by temperature, electromagnetic field and chemical environment. The latter is achieved by reshaping the geometry or configuration of constitute elements *via* external conditions. Both the material response and geometry can thus affect the optical chirality of the system.

Taking advantage of light strength-dependent conductivity of silicon, Zhang *et al* achieved an adaptable optical chirality in THz regime *via* modification of the electric-magnetic coupling, as shown in **Figure 8a**.[133] By employing the similar idea, Yin *et al*

realized an effective modulation of optical chirality in mid-IR region using a phase change material (PCM) $Ge_3Sb_2Te_6$ (GST-326). As shown in **Figure 8b**, when the phase of GST-326 transits from amorphous phase to crystalline phase, great change in the dielectric environment modulates the optical chiral response of layered plasmonic nanoantenas.[134] By employing the permittivity change of magnesium (Mg) under different hydrogen (H_2) concentrations, Duan *et al* accomplished an efficient CD modulation, as shown in **Figure 8c**.[135] It is noteworthy that structures in all three works are C4 symmetric, which verifies that CD change is solely a consequence from structural chirality alteration.

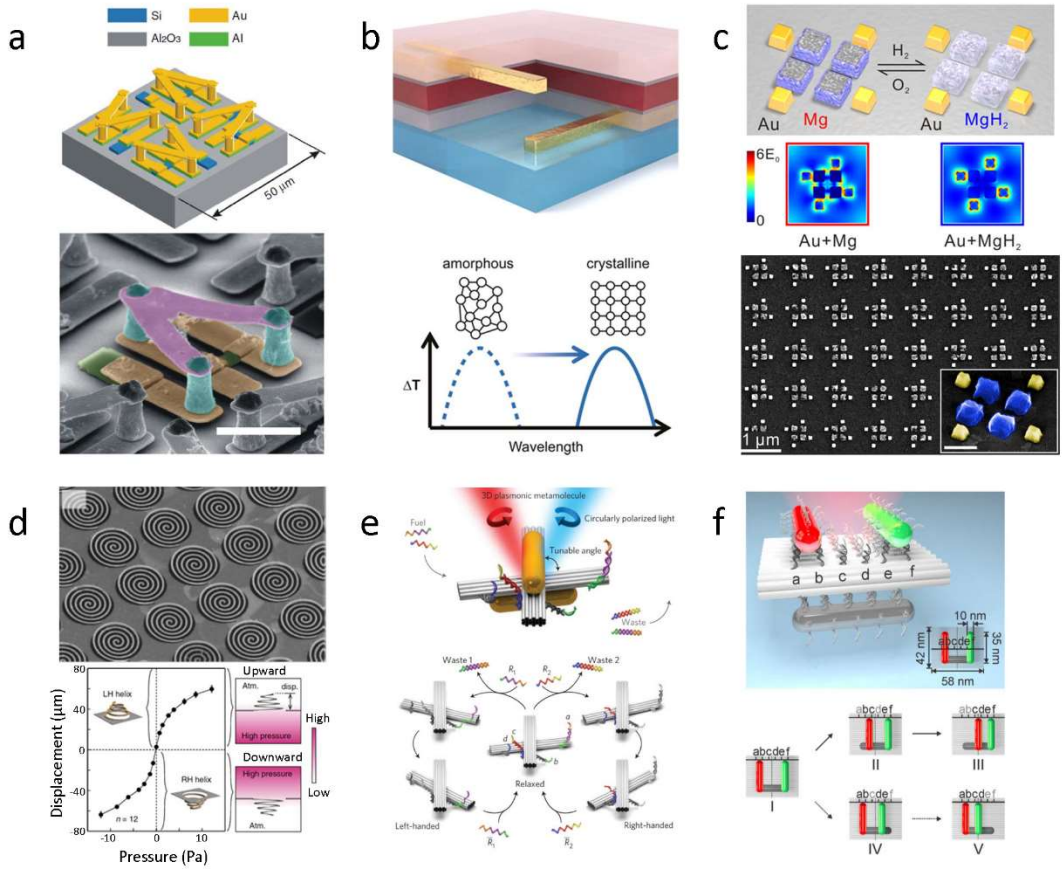

Figure 8. Active 3D chiral structures. (a) Schematic and SEM images of double-layered active chiral structure. This structure can be tuned by extra light field to change the THz CD. (b) Thermally induced active chiral structure based on GST-326, whose material properties can also be electrically and optically switched. (c) Hydrogen-regulated active chiral structure. (d) Mechanically induced active chiral structure based on MEMS. (e) DNA-regulated chiral nanostructure. (f) Chiral nanostructure with nanorods can actively "walk" on a DNA plane. Reproduced with permission, Zhang et al.[133] (a) Copyright 2012 Springer Nature. Reproduced with permission, Yin et al.[134] (b) Copyright 2015 American Chemical Society. Reproduced with permission, Duan et al.[135] (c) Copyright 2016 American Chemical Society.

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Since chirality is a geometric property, alteration of geometry can efficiently modulate structural optical chiral response. Thus, flexible material could be an excellent candidate for designing reconfigurable structures. Taking advantage of modest hardness of gold, Kan *et al* designed a structure with flexible gold spiral fabricated in micro holes on a silicon substrate. Stretch direction of the gold spiral was controlled by barometric changes in the two flanking space. Opposite chirality observed under opposite stretch directions further proved that this nanostructure is indeed geometry adaptable, as shown in **Figure 8d**.[136] Limited by elasticity of metal materials, it is almost unfeasible for this structure to be reduced to optical regime. A similar design was realized in microwave regime and was used to tune the nonlinear responses by Slobozhanyuk *et al*.[139]

Schreiber *et al* employed DNA nano-helices assembled nanocluster of spheres as a chiral structure, since the structural direction can alter accordingly with its distributed environment.[140] When placed in buffer, DNA molecules of the structure become vertical to surface. In contrast, when the buffer is removed and the surface dries up, DNA nano-helices bow down into parallel direction with the surface. The optical chiral responses are significantly different under these two conditions, similar to the case shown in **Figure 1b**. Taking reconfigurable DNA origami template consisting of two helix bundles as prototype, Kuzyk *et al* further attached a gold nanorod to each of the two helix bundles to achieve chiral structure. Since optical chiral response is dependent on angle between two helices, and the helical angle is determined by DNA molecules at their ends, modulation of optical chiral response can be reached by changing the attached molecules (practically immerse the structure in solution with different molecules). Corresponding structure is shown in **Figure**

8e.[137] Furthermore, the authors accomplished a variant design using ultraviolet and visible light illumination as an input trigger to modulate angle between helix bundles. The gold nanoparticle is either chiral or achiral according to whether the two DNA helices are in transcis photoisomerization locking or unlocking states.[141]

Zhou *et al* introduced another design implementing DNA origami with individually addressable sites.[142] Two gold nanorods were vertically attached to the DNA origami, with one rod being static and the other one being capable of walking over the surface with the help of fuel DNA stands. Thus by manipulating movements and arrangements of the two nanorods, chirality of the system can be switched from left to right and back accordingly. As the structure changes, its CD spectra showed significant alteration and finally reach a completely reversal spectrum compared to the original one. Through a more complex experiment design, the authors also proved that alteration of optical response was solely ascribed to structure and arrangement changes. In summary, the authors have demonstrated the feasibility of optical manipulation with external stimulus, and proposed an efficient way to move two individually addressable walkers on DNA origami.[138]

Active chiral structures are of enormous practical potential. One of their most prospective applications is to serve as modulators. Rapid development of communication and holography technology demand urgent requirement for modulators that can tune light strength and phase more efficiently. Versatile modulators for light strength, phase and polarization can be realized by integrating traditional polarizer or wave plate with various components including electric, magnetic and light filed regulatory active optical chiral media. Another application field is sensor development. Components that can regulate optical chirality should also be detected using similar mechanism. For example, studies in Ref. [135] can be applied as sensor to determine concentration of hydrogen, studies in Ref. [136] can be used to detect pressure or sound. Nano-walker made of DNA molecules can serve as micro or nano-motor, in which case chirality should be responsible to measure and monitor working status or nano-

motors. The chiral nanostructures could have practical applications in bio-sensing. For example, Gao *et al* [143] and Li *et al* [144] measured the existence of chiral molecules by the changing of CD spectrum of chiral nanostructures in living cells.

5. Conclusion and future perspectives

Chirality is a geometric property, the strength of which can be determined by molecule or nanostructure symmetry. Molecules or subwavelength structures with chirality always possess chiral response. However, geometric chirality does not directly correspond to optical chirality. In this review, we firstly analyzed structural requirements for generating optical response, thus concluded several important design concepts. The first concept is to generate electro-magnetic coupled mode through nonorthogonal electric and magnetic resonances. Another concept is to construct two nonparallel coupled electric dipoles at different equiphase planes. Coupling between two resonances can be achieved *via* either direct connection or indirect near-field. A careful review of previous works revealed that most nanostructures with strong optical response can be categorized into these concepts.

Until now, large amount of endeavor have been invested in developing new 3D chiral nanostructure fabrication techniques, including 3D direct laser writing, multi-layered lithography, glancing angle deposition and electron beam-induced deposition. These techniques are not only effective in fabricating chiral nanostructures, but also applicable in manufacturing other complex 3D nanostructures, laying a solid foundation for further studies in nanoscience.

Well-achieved optical chiral nanostructures have proved their great application values, including the most fundamental circular polarizer, early-proposed negative index, and the more complex nonlinear optical device, sensor and optical modulator. As to interdisciplinary research, optical chirality also plays an important role, for instance, in nano-motors composed of DNA molecules.

Current studies on optical chiral nanostructures are prospective, but more works still need to be performed in the future. From the theoretical point of view, previous designs are mainly based on geometric chirality, thus more comprehensive physics understanding and following structure design can be further improved. In addition, most realized optical chiral nanostructures designs using LSPRs have disadvantages in Q-factor and loss. Attempts with different kinds of resonance, such as Mie resonance and lattice resonances can also be conducted for optical chiral response enhancement. As to nanofabrication methods, current techniques are quite sophisticated in precision and 3D effect, but limited in cost control and product area. Refinements are still in need before optical chiral nanostructures can really benefit practical applications. In addition, more endeavors are worthy devoting to bringing chiral nanostructures research together with optical, physics, chemistry and biology studies, including quantum levels, active molecules and biomacromolecules. We enthusiastically envision devices with practical application values fruited from such interdisciplinary research.

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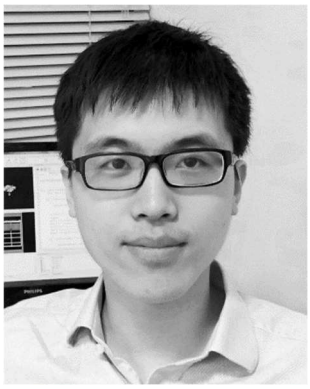

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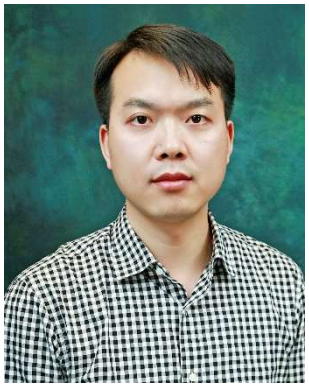

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Three-dimensional metaphotonic structures with intrinsic chirality are able to support optical chiral response, including both optical rotation and circular dichroism, orders of magnitude higher than their natural occurring counterparts do. They have drawn increased attentions due to their potentials in fundamental researches in physics, chemistry and biology, and practical applications, such as negative refractive index, molecule sensor and modulators.

Keyword: chiral, plasmonic, nanofabrication, metamaterial, nano-optics

Author: M. Qiu, L. Zhang, Z. Tang, W. Jin, C.-W. Qiu* and D. Y. Lei*

Title: Three-dimensional metaphotonic nanostructures with intrinsic chirality

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